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DISTILLATION SYSTEM WITH INDIVIDUAL FRACTIONATION  
TRAY TEMPERATURE CONTROL

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BACKGROUND OF THE INVENTION

1. Field of the Invention

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The present invention is directed to  
distillation systems having columns with  
individual trays having separate and distinct  
heating and/or cooling capabilities, and in some  
preferred embodiments, to such systems having  
tray cooling units which rely upon phase change  
coolant injection. Preferred embodiments also  
include preprogrammable and programmable systems.

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2. Information Disclosure Statement

The following patents are representative of  
prior art related to various types of

heated/cooled reactors, distillers and the like:

U.S. Patent No. 1,744,421 to W.F. Stroud,  
Jr., et al. describes a process which comprises  
passing vapors upwardly through a plurality of  
5 fractionating zones at different levels, passing  
reflux liquid downwardly in a continuous stream  
through said zones countercurrent to and in  
contact with said vapors, withdrawing liquid from  
one of said zones at a rate in excess of the rate  
10 at which reflux liquid is passed there-through,  
cooling the liquid so withdrawn, and returning  
liquid so cooled to a zone at least as high as  
the zone from which it was withdrawn.

U.S. Patent No. 2,739,221 to Glen H. Morey  
15 describes a vessel heater as recited in which  
includes a first valve communicating with a

supply of non-inflammable and non-combustion-  
supporting fluid in its gaseous phase to regulate  
admission of a quantity of fluid to blanket said  
heating element and thereby preclude ignition of  
combustible products adjacent said heating  
element, and a second valve communicating with a  
supply of non-inflammable and non-combustible-  
supporting fluid in its liquid phase to regulate  
admission of a quantity of fluid to effect rapid  
cooling of the vessel heater.

U.S. Patent No. 2,894,881 to Clinton M.  
Wolston, Jr. et al. describes a laboratory  
distillation testing apparatus having a condenser  
tank, a flask, a flask supporting means, a  
heating means, a condenser tube passing through  
the said tank, and a light diffusing panel, the

improvements which comprise a recess in said  
condenser tank, a shield means disposed within  
said recess, adjustable shelf means carried by  
said shield means for supporting said flask,  
5 conduit means below said tank, and solenoid valve  
means on said conduit means, the discharge end of  
said conduit means projecting forwardly of the  
rear wall of said recess below said condenser  
tube inlet and arranged to discharge forwardly  
10 and downwardly towards said shelf means.

U.S. Patent No. 3,133,014 to Willis J.

Cross, Jr. describes in the fractionation of a  
synthetic crude effluent obtained from a  
hydrocarbon conversion reaction, wherein said  
15 effluent is fractionated by introducing the same  
at least partially in vapor state into a

fractionating tower for separation into several cuts including (a) a gasoline-containing cut and (b) a bottoms product having a mid-boiling point above 600 F., and wherein a portion of said

5 bottoms product is cooled and admixed in liquid state for direct heat exchange with the hot vapor effluent directed to the fractionating tower; the improvement which comprises introducing said portion of cooled liquid bottoms axially into  
10 the vapor conduit carrying the flowing stream of said vapor effluent at a location up-stream of said fractionating tower, said liquid being introduced by discharging a circumferentially expanding cone of said liquid concurrently into  
15 said flowing vapor stream, and said location being so selected that the expanding cone of

liquid does not contact the inner wall of said conduit along a substantial portion of the path of flow of said liquid toward said tower.

U.S. Patent No. 3,143,167 to Adolf Vieth

5 describes a temperature controlled enclosure comprising, a first metal wall surrounding the enclosure space, a heating means in thermal contact with said first wall for raising the temperature of the enclosure, a second metal wall

10 surrounding the heating means, cooling means in thermal contact with said second wall for lowering the temperature of the enclosure, a first temperature-sensitive element in thermal contact with said first metal wall, a second

15 temperature-sensitive element in thermal contact with said second metal wall, and a control

circuit connected between said elements and said  
heating and cooling means for energizing the  
heating and cooling means selectively to produce  
a desired temperature within the enclosure, said  
5 control circuit including a bridge, an amplifier,  
and a switching means for connecting the heating  
means to a source of power when said first  
temperature-sensitive element is connected to the  
bridge and for activating the cooling means when  
10 said second temperature-sensitive element is  
connected to the bridge.

U.S. Patent No. 3,239,432 to Joseph C.

Rhodes et al. describes an apparatus for  
controlling the operation of a first distillation  
15 column and for determining the distillation  
properties of a product sample from said first



column which apparatus comprises: means for  
withdrawing a product sample containing a mixture  
of liquids having different boiling points from  
said first column; a test column member; a  
5 plurality of liquid-retaining trays spaced apart  
vertically within said test column; a liquid  
sample container positioned below said test  
column and in flow communication with the bottom-  
most portion of said test column; means for  
10 receiving said withdrawn product sample and  
introducing a known amount of said product sample  
into said container; means for vaporizing liquid  
sample introduced into said container; vapor  
riser means for passing vapors from the lower  
15 portion of said test column upwardly through said  
test column to intimately contact liquid

retaining on said trays; condensing means communicating with the upper end of said test column to condense all the vapors rising from the upper-most of said trays; means for returning the resulting condensate to the upper-most of said trays; means for maintaining the test column pressure at a substantially constant pressure during a run; means for maintaining a pre-selected level of liquid on said trays; temperature sensing means to sense the temperatures and produce a temperature signal indicative thereof of equilibrium vapors above the trays in said test column; means for receiving said temperature signal and correlating the sensed temperatures with the distillation properties of a known product sample of

approximately the same composition as said sample  
being run and produced an output signal relative  
to said correlation; and means for receiving said  
output signal and adjusting the control  
5 parameters of the first column in accordance with  
said output signal.

U.S. Patent No. 3,502,547 to R.E. Bridgeford  
describes a separation system for the recovery of  
a middle boiling fraction from a feed mixture  
10 containing said middle boiling fraction and  
higher and lower boiling fractions, comprising:

(a) a single fractional distillation column  
containing:

(1) a top section, and

15 (2) a bottom section, said bottom

section having a smaller diameter than

said top section;

(b) means for passing said feed mixture  
into said top section;

(c) a solid vapor impermeable plate  
5 separating said top section from said bottom  
section and having as the only fluid passageway  
through said plate at least one downcomer;

(d) a tray positioned as the top tray in  
the top portion of said bottom section and spaced  
10 below said plate to form a vapor space  
therebetween, said tray having weir means for  
maintaining a liquid level thereon, said at least  
one downcomer extending downwardly into the  
liquid retained on said tray so that said at  
15 least one downcomer passes only liquid from the  
bottom portion of said top section to the top

portion of said bottom section while vapor is prevented from passing upwardly from said bottom section to said top section;

(e) means for removing from said fractional distillation column and from the system a primary overhead product stream comprising said lower boiling fraction by removing an overhead vaporous fraction from the top portion of said top section;

(f) means for removing from said fractional distillation column and from the system an intermediate vaporous product stream comprising said middle boiling fraction by removing a vaporous fraction from the vapor space above said tray in the top portion of said bottom section;

(g) means for removing from said fractional

distillation column and from the system a bottom product stream comprising said higher boiling fraction by removing liquid from the bottom portion of said bottom section;

5 (h) reboiling means for heating liquid in the bottom portion of said top section to provide stripping vapor for said top section; and

(i) reboiling means for heating liquid in the bottom portion of said bottom section to provide stripping vapor for said bottom section.

10 U.S. Patent No. 3,544,428 to Marvin E. Mellbom describes a fractionation apparatus comprising a vertically elongated chamber having an upperflashing section, and upper stripping section disposed externally of said elongated

15 chamber and connecting with said upper flashing

section and having direct liquid communication  
with said upper flashing section, a lower  
flashing section in said chamber below said upper  
flashing section, a lower stripping section  
5 connected to the lower portion of said lower  
flashing section and having fluid communication  
therewith, said lower and upper flashing sections  
in said chambers being separated by a transverse  
partition adapted to pass vapors from the lower  
10 section into said upper section and to prevent  
liquid flow therebetween, a common fractionating  
section above said upper flashing section, a  
conduit means extending upwardly from said  
partition to said upper flashing section, the  
15 upper end of said conduit means being spaced  
above the elevation of the hereinbelow specified

first feed inlet, said conduit means providing  
vapor communication between said flashing  
sections, a first feed inlet for introducing a  
first heated hydrocarbon feed into said upper  
flashing section, a second feed inlet for  
introducing a second heated hydrocarbon feed into  
said lower flashing section, said first feed  
being independent of and having a different  
composition than both said second feed and the  
hereinbelow specified second bottoms fraction,  
stripping inlet means for introducing stripping  
material into each of said stripping material  
into each of said stripping sections, at least  
one product outlet in the fractionating section  
of said chamber, a bottoms fraction outlet in  
said upper stripping section for withdrawing a



first bottoms fraction therefrom, and a bottoms  
fraction outlet in said lower stripping section  
for withdrawing a second bottoms fraction  
therefrom.

5 U.S. Patent No. 4,117,881 to Williams et al.

describes blood cells, blood marrow, and other  
similar biological tissue is frozen while in a  
polyethylene bag placed in abutting relationship  
against opposed walls of a pair of heaters. The  
10 bag and tissue are cooled with refrigerationing  
gas at a time programmed rate at least equal to  
the maximum cooling rate needed at any time  
during the freezing process. The temperature of  
the bag, and hence of the tissue, is compared  
15 with a time programmed desired value for the  
tissue temperature to derive an error indication.

The heater is active in response to the error indication so that the temperature of the tissue follows the desired value for the time programmed tissue temperature. The tissue is heated to compensate for excessive cooling of the tissue as a result of the cooling by the refrigerating gas. In response to the error signal, the heater is deactivated while the latent heat of fusion is being removed from the tissue while the tissue is changing phase from liquid to solid.

U.S. Patent No. 4,276,264 to Redikultsev et al. describes a device for sterilizing water-containing liquid media by steam which comprises a sterilizing vessel with inlet and outlet connections for processed liquid media. A heater is provided in the lower portion of the vessel,

while a condenser is arranged in the upper  
portion thereof. The vessel also houses a  
coaxially mounted steam-transfer unit  
representing gas-lift tube with a diffuser  
5 disposed over the heater.

U.S. Patent No. 4,346,754 to Imig et al.  
describes a heating and cooling apparatus capable  
of cyclic heating and cooling of a test specimen  
undergoing fatigue testing. Cryogenic fluid is  
10 passed through a block 10 clamped to the specimen  
11 to cool the block and the specimen. Heating  
cartridges 13 penetrate the block 10 to heat the  
block and the specimen 11 to very hot  
temperatures. Control apparatus 36 and 46 is  
15 provided to alternately activate the cooling and  
heating modes to effect cyclic heating and

cooling between very hot and very cold temperatures. The block 10 is constructed of minimal mass to facilitate the rapid temperature change thereof.

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U.S. Patent No. 4,714,542 to William

Lockett, Jr. describes a distillation vapor and feed mixing and subsequent separation process and apparatus involving the introduction of a vaporizing liquid feed into a flashing zone via a tangential nozzle into a mixing and separation chamber which directs the feed into a circumferential path to enhance mixing, and the redirection of rising vapors from the distillation below the flash zone by baffling these vapors into the chamber inlet. The rising vapors are inspired by the high velocity feed

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at the inlet side of the chamber and intimate contact and mixing of the rising vapors with the vaporizing feed are enhanced by creating a spinning action. Preferably, the chamber runs peripherally and slightly downward along the inside wall of the distillation column along an arc no greater than 360. Alternatively, the mixing section of the mixing and separation chamber may be located outside of the distillation tower and the feed, passing through a jet ejector would inspire the rising vapors. Increasing contacting and mixing efficiency in a distillation flash zone increases the yield of more valuable overhead product for the same energy input or permits lower energy input for constant separation between overheads and bottoms

in the flash zone.

U.S. Patent No. 5,326,436 to Sampath et al.

describes a method of feeding a fractionator feed  
mixture having a wide-boiling range vapor-liquid  
mixture is provided. Also, provided is a  
fractionator feed section adapted to receive a  
two phase feed mixture and has operational  
stability when fed a feed mixture which generates  
significant volume of vapor in the feed section.

U.S. Patent No. 6,193,849 to William

Lockett, Jr. describes a fractionator having a  
fractionation vessel, a reactor effluent vapors  
inlet, a vapor feed contacting zone, a baffled  
contacting section above the vapor feed  
contacting zone, a tops section above the baffled  
contacting section, a heavy bottoms liquid hold-

up pool section below the vapor feed contacting zone, a bottoms outlet, a bottoms recycle system with a heat exchanger. Recycled, cooled bottoms is fed back to the fractionation vessel at the heavy bottoms liquid hold-up pool section and above the vapor feed contacting zone. The improvements involve providing a separation tray and downpipe for separating cooler bottoms liquid from hotter product vapors within the fractionation vessel: to avoid condensation and absorption of product vapors by the liquid pool; to have more rapid and uniform quenching of hot liquid entering the pool; and substantially reduce costly onstream maintenance to clean fouled bottoms recycle exchangers.

Notwithstanding the prior art, the present

invention is neither taught nor rendered obvious thereby.

SUMMARY OF THE INVENTION

5           The present invention distillation system  
with individual fractionator tray temperature  
control, with the use of either a heating element  
or a cooling element, and in some preferred  
embodiments, the use of both a heating element  
10           and a cooling element in a plurality of  
fractionator trays.

          In the broadest sense, the present invention  
system includes at least one distillation column  
having a plurality of fractionation trays, and  
15           having feed input means, liquid removal means,  
and vapor removal means, wherein at least a



majority of the plurality of trays include at least one of a heating element and a cooling element. Control means is also included for separate control of each of the heating  
5 element(s) and/or said cooling element(s) for the trays having at least one of a heating element and/or a cooling element.

In some preferred embodiments, the distillation system of the present invention  
10 includes a plurality of distillation columns functionally connected to one another. For example, there may be a specified number of distillation columns and each distillation column after a first column has a vapor removal means  
15 from a preceding distillation column connected to its feed input means, so as to create an in

series system.

In some embodiments, The distillation system the majority of the trays have a heating element and/or a cooling element which is integrally formed within the tray.

In some embodiments, the cooling element may be a refrigeration unit, a coolant heat exchanger, or other type of cooling element, or it may be a phase change coolant element.

The present invention distillation system preferred cooling element is a phase change coolant element which may include at least one open area within a tray, with the cooling element having an inlet port for injection of a coolant into the open area within the tray. (This open area acts as a heat absorbent area). The cooling

element will also include an outlet port for removal of phase change coolant in gaseous state from the open area of the tray.

These phase change cooling elements of the present invention preferably also includes injection means for injection of the phase change coolant in liquid form into the inlet port of the cooling element for creation of cooling by phase change from a liquid state to a gaseous state.

In most preferred embodiments, the present invention distillation system further includes control means connected to the cooling elements and the heating elements for programmable automatic control therefore, e.g. for control of injection means to control at least one of on/off flow and rate of flow, and to control at least

one of on/off heating and rate of heating. The control means may include a programmable device, with appropriate software.

The present invention distillation system with control means preferably includes software in the system for an injection means physical control device, for cyclical on/off control thereof to establish at least one predetermined temperature sequence involving a plurality of diverse, programmable temperature levels, with means to vary on/off time sequences.

Typically, when a phase change liquid coolant cooling element is included, a remote reservoir of a phase change coolant is connected to the injection means and inlet port wherein the reservoir contains a phase change coolant in a

liquid state under pressure. It may include phase change coolant generation means connected thereto.

When a phase change coolant is included it may preferably be an environmentally inert material which absorbs heat upon vaporization and has a boiling point below room temperature at atmospheric pressure. Such a phase change coolant may be selected from the group consisting of inert gases, carbon dioxide and nitrogen.

In some embodiments, the present invention distillation system further includes at least one membrane cartridge located in a column between trays for membrane filtration, and typically utilizes a plurality of these membrane cartridges to enhance material separation in the process.

In some embodiments, a downstream vacuum removal unit is located on a last column vapor removal means to enhance separation of materials.

Further, there may be included at least one

5 upstream liquid combination unit having plural

liquid source inlet means, selective controls for

operating any one or more of the plural liquid

source inlet means, and outlet means. These are

connected, either directly or indirectly, to a

10 first column in the present invention system.

When indirectly connected, there may be an

evaporation chamber located between the liquid

combination unit and the first column and

connected to the liquid combination unit outlet

15 means and to a feed input means of the first

column to evaporate the liquids into gaseous

phase or partial gaseous phase for input to the columns.

There may be at least one valved recycle line connected between two columns to selectively recycle at least a portion of liquid from a downstream column back to an upstream column.

In order to more closely regulate tray temperatures a plurality of temperature sensing means may be located at various tray levels for sensing temperature of the tray levels, and are connected to said control means for temperature feedback to computer software for readjustment of the heating units and said cooling units to fine tune the achievement of predetermined temperatures.

The heating units of the present invention

may be any known heating element, but is preferably a heat exchange system or a heating coil system.

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BRIEF DESCRIPTION OF THE DRAWINGS

The present invention should be more fully understood when the specification herein is taken in conjunction with the drawings appended hereto wherein:

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Figures 1 and 2 illustrate different types of trays used in the present invention system;

Figure 3 shows a detailed schematic diagram of a single tray in a present invention system, showing controls; and,

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Figure 4 illustrates a detailed embodiment of a present invention system.



DETAILED DESCRIPTION OF THE PRESENT INVENTION

5           The present invention distillation system is  
taken to mean a distillation system which  
involves at least one distillation column which  
includes the other features of the invention set  
10       forth above and below. The term "distillation"  
and "fractionation" should be interpreted broadly  
to mean the process of separating liquid  
materials of differing boiling points by the  
15       vaporation and condensation processes. The  
invention set forth generically in the summary is  
illustrated by the description of the appended  
drawings. Thus, the following descriptions  
should not be deemed as strict interpretations of  
the present invention.

Referring now specifically to Figure 1,

there is shown a portion of a present invention  
system 1 which includes distillation column 10  
and a fractionation tray 3. Tray 3 includes  
both a controlled heating unit and a controlled  
cooling unit, critical features of the present  
invention. As mentioned above, a tray may  
include either a heating element or a cooling  
element or both, but in this embodiment, both are  
illustrated. In addition, a heating unit 5 is  
included which, in this embodiment, is a heating  
coil it includes wire connection to a controller,  
preferably a programmable microprocessor.  
Further details of operation are set forth in  
conjunction with the description of Figure 3  
below.

Tray 3 of Figure 1 includes an inlet port 7

for input of a coolant, open area 9 for absorbing  
heat and outlet port 11. This cooling unit open  
area 9 may be an annular space or may have some  
other configuration, but is designed to have  
5 significant surface area between it and tray 3 to  
enhance heat absorption. The coolant may be a  
phase change coolant and outlet port 11 will  
preform as a gaseous state exhaust port. Inlet  
port 7 and outlet port 11 extend into  
10 distillation column 10, as shown. Conventional  
features such as the vapor uptake 15, bubble cap  
17 and downflow pipe 19 are also included.

Referring now to Figure 2, there is shown in  
alternative present invention tray 20 which  
15 includes vertical orifices 21, 23 and 25,  
throughwhich liquid may flow downwardly and

vapors may flow upwardly wherein liquid is contained within the recess 27. Tray 20 includes a set of 4 circular bores which are shown as 29-29', 31-31', 33-33' and 35-35'. Inlet and outlet connections are located on the back side of tray 20 and are not shown. Each of the aforesaid bores may be used as a cooling element or as a heating element. Thus, tray 20 may include only a heating element with all 4 bores being connected to an elevated temperature liquid coolant material, or all 4 bores could act as only a cooling element and be connected to a cooling source. Alternatively, one or more could be connected to a cooling source and one or more could be connected to a heating source so as to create a tray 20 which provides both a heating

and cooling in a single tray. Tray 20 could be included on one or more columns and could constitute a plurality of trays located in a column to provide distillation and fractionation temperature controls previously unachievable.

Figure 3 shows a schematic diagram in its simplest form illustrating a present invention system 100. It includes phase change coolant supply 115, feeder line 117, injector control 111 and injector 109. Injector 109 is connected to a tray with a cooling unit inlet port (not shown) contained within tray 130. Tray 130 includes both a cooling element and a heating element. On the outlet side is outlet port 129 for removal of coolant in its gaseous form. Typically, it is injected in liquid form. Programmable

microprocessor 121 is connected to both the injector control 111 via wiring 123 and the heating element via wiring 125. In an actual system, tray 130 would be duplicated repeatedly in one or more columns and would be connected to the programmable microprocessor so that many tray temperatures could be controlled in real time and/or preprogrammed.

Additionally, preprogrammable microprocessor 121 is connected to temperature sensor 135 for microcontrol and feedback to preprogrammable microprocessor 121 in preferred embodiments.

Figure 4 shows an overall present invention distillation system 200 with 3 distillation columns 201, 203 and 205. Column 201 includes the feed input line, 211 a vapor removal line and

a liquid removal line 215. Similarly, columns  
203 and 205 have input feed lines 221 and 231,  
vapor removal lines 223 and 233 and liquid  
removal lines 225 and 235. In this embodiment,  
5 there are also connections and valving to permit  
recycling from columns 203 and 205 back to  
upstream column(s), as shown. Column 201, 203  
and 205 include a plurality of trays similar to  
the trays shown in Figures 1 and 2 above with the  
10 heating and cooling elements contained therein.  
Coolant supply and heating supply 237 and 239 are  
connected to the columns 201, 203 and 205 and are  
merely illustrated as being schematically  
connected to column 201. Additionally, they are  
15 controllably connected to the computer system  
shown as microprocessor 250, keyboard 251 and

monitor 253.

Three input lines 241, 243 and 245 are provided to receive mixtures of liquids for separation and valves to 255, 257 and 259 are provided to control flows to permit shutoff, input to liquid combination unit 291 or bypass of any or all of these lines. For example, mixtures to be separated are fed into liquid combination unit 291, via line 293 are transferred to evaporation chamber 295 and subsequently fed into first column 201 via vapor removal line 211. In this embodiment, separation membrane cartridges such as cartridge 261 and 263 in column 205 are included to further enhance constituent separation. Bottoms may be removed via liquid removal lines 215, 225 and 235, and outlet lines



275, 277 and 279. Evaporative materials are removed via line 261 to cooling chamber 271 connected to vacuum pump 273 with outlet 281 and recycle lines 283 and 285.

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The above described systems of the present invention may be constructed of any size and arrangement convenient for a particular purpose. Therefore, the invention could be embodied in the form of very tall, full scale columns towering 50 or 100 feet in height or the invention could be embodied in the form of a bench lab setup. In a preferred embodiment, the present invention may be in the form of a portable system useful for collection of biochemical waste and solvent for recovery and reuse of the solvent.

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The present invention systems may include

programmable temperature/time sequences utilizing  
a microprocessor for the heating and cooling  
unit. With this system, various distillation  
requirements are automatically achieved, such as  
5 heating/cooling, cooling/heating sequences,  
refluxing and condensing. The preferred  
embodiments of present invention system cooling  
unit uniquely relies upon phase change coolants  
where the endothermal heat of evaporation is  
10 absorbed from the tray when the phase change  
coolant is injected into the heat absorbing area  
with a programmable device, e.g. a computer,  
controlled injector. Environmentally inert  
phase change coolants are utilized and evaporated  
15 and dissipated to the atmosphere in gaseous form.

The tray utilized in the present invention

may be any form of tray used in  
distillation/fractionation and is capable of  
transmitting heat therethrough to add or remove  
heat during a distillation process. Thus, the  
5 tray may be glass, ceramic, cement, metal or  
other material. It will have connected thereto  
(inside, outside, both or embedded) at least one  
temperature sensor, e.g. a thermocouple, to sense  
temperature. It preferably has at least two  
10 temperature sensors, for example, one at an upper  
portion of the tray and one at the lower portion  
thereof. The temperature sensors are connected  
to the control means, which has a programmable  
device, e.g., a computer, a microprocessor or  
15 other known devices as its central component.

The heating unit is one which may be

automatically controlled, either by off/on  
sequencing or amount of heating (rate) or both.  
The heating unit may be conductive, convective,  
radiant, directly or indirectly, e.g. by heat  
5 exchanger or combination of heating mechanisms  
but is typically an exchanger heating element or  
an electric heating element type unit, e.g., with  
electrical convection, controlled by the  
microprocessor.

10           The heating unit and cooling unit may be in  
close proximity to one another or spaced apart  
substantially depending upon the actual design,  
conditions and results desired.

15           The cooling unit of the present invention,  
like its heating unit counterpart, may take on  
any physical shape to accommodate the heat

transfer (removal for cooling) relative to the  
tray. The preferred cooling unit of the present  
invention system includes a cooling element with  
an inlet port, a heat absorbing area and an  
outlet port or a plurality of one or more of  
these components. It also includes injection  
means at the inlet port for controlled injection  
of phase change coolant. While the present  
invention system may be manufactured and sold in  
various configurations without a phase change  
coolant supply, in actual use a phase change  
coolant supply is essential, e.g. by attachment  
of one or more pressurized inert liquid tanks or  
with a generator, or a compressor or other  
coolant creating, compressing or storing means.

The cooling element may be coiled tubing or

a molded, machined or an otherwise-formed open area within the tray to permit injection of phase change coolant. In other words, the open area of the cooling element is enclosed, e.g. with materials of construction which preferably include heat conductive characteristics. The phase change coolant is injected into the heat absorbing area at the inlet port and evaporates under normal pressure to its gaseous state and exhausts as gases through the outlet port. It is the endothermic heat of evaporation to the phase change coolant that absorbs heat from the tray to effect cooling.

The phase change coolant may be any material which evaporates below the desired temperature of the area to be cooled, e.g., below 24° C, and

preferably, below 0° C. Such materials are liquid under pressure and may be stored as such in storage reservoirs, e.g. tanks, for subsequent use or otherwise provided as described above.

5 These coolants go through at least one phase change to effect a neat heat absorbing transition, are environmentally inert, i.e. harmless to the environment when dissipated, and include such phase change coolants as are  
10 presently and/or will become commercially available. They include, but are not limited to, the elements known as inert gases, carbon dioxide, nitrogen, etc.

The cooling mechanism of the preferred  
15 embodiment of the present invention is based on the heat exchange during the phase change coolant

material. A precise heat exchange control can be readily achieved by an appropriate selection and adjustment between either liquid to gas or a sequential phase change of liquid to solid then solid to gas. Commonly used in coolants are pressurized liquid carbon dioxide, or pressurized liquid argon, or pressurized liquid nitrogen. Pressurized liquid materials will be selected specifically to accomodate the desired critical point, depending upon the particular materials being separated in the column.

The injection means will typically include an injection nozzle, such as a stainless steel nozzle, a valving mechanism and a supply line, with the valving mechanism directly upstream from the injection nozzle. (In cases where smaller



diameter tubing or inlet means is used, then such tubing or inlet means may act as the nozzle itself, without added hardware.) The valving mechanism may be a flap or shutter valve or other on/off valve, or it may be a controlled opening (flow rate controlling valve) such as a stem valve or gate valve. The on/off valve mechanisms may be opened and closed by solenoids or switches or other known devices, and the flow controlling valves may be opened and closed by servo-drivers or other rotating or lifting devices. In a more complicated system, both types of valves, i.e. on/off and flow rate controlling valves may be used to offer both types of controls in the system.

The control means is any programmable

device, such as manual switches, dials, buttons,  
levers, etc., with sensors for feedback, a  
computer or microprocessor with appropriate  
software or sequence input, external inputs and  
5 wiring to the cooling unit, to the heating unit  
and preferably, to the tray.

More specifically, the programmable device  
may have output information available to a user,  
e.g. a microprocessor may have a display which  
10 includes a readout and programming inputs. For  
example, it could have a plurality of buttons,  
input means, selection means, switched, keypads,  
etc., with choices including "COLUMN NUMBER",  
"TRAY NUMBER", "SEQUENCE NUMBER", "TEMPERATURE",  
15 and "TIME" with a numerical keyboard, and the  
microprocessor itself will divide when to use the

heating unit and when to use the cooling unit to achieve the programmed temperatures for the specified times. The "TIME" inputs could be elapsed time needs or actual clock start and end times. In a more preferred embodiment, additional buttons, controls, inputs, icons, selections, etc. could include "HEATING UNIT" and "COOLING UNIT" selections so that both units could operate simultaneously or separately or both, as the user may desire other control inputs/outputs should now be evident to the artisan. Pluralities of these will be provided to accommodate each temperature controlled tray in the system. In yet another embodiment, a user may be offered the opportunity to select proportional controls for flow, tolerances from a

predetermined set of choices and other  
parameters, as a designer may offer to end users.  
Also, the programmable device may have real time  
override capabilities, as well as time delay  
5 input capabilities before start-up is initiated  
or even offer unlimited off sequences between  
heating and/or cooling sequences for inputted  
periods of time. Other programming possibilities  
should now be apparent to the artisan without  
10 exceeding the scope of the present invention.

Obviously, numerous modifications and  
variations of the present invention are possible  
in light of the above teachings. It is therefore  
understood that within the scope of the appended  
15 claims, the invention may be practiced otherwise  
than as specifically described herein.